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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE

No. 1488

COMPARISON OF CRYSTAL STRUCTURES OF 10 WROUGHT
HEAT RESISTING ALLOYS AT ELEVATED
TEMPERATURES WITH THEIR CRYSTAL
STRUCTURES AT ROOM TEMPERATURES

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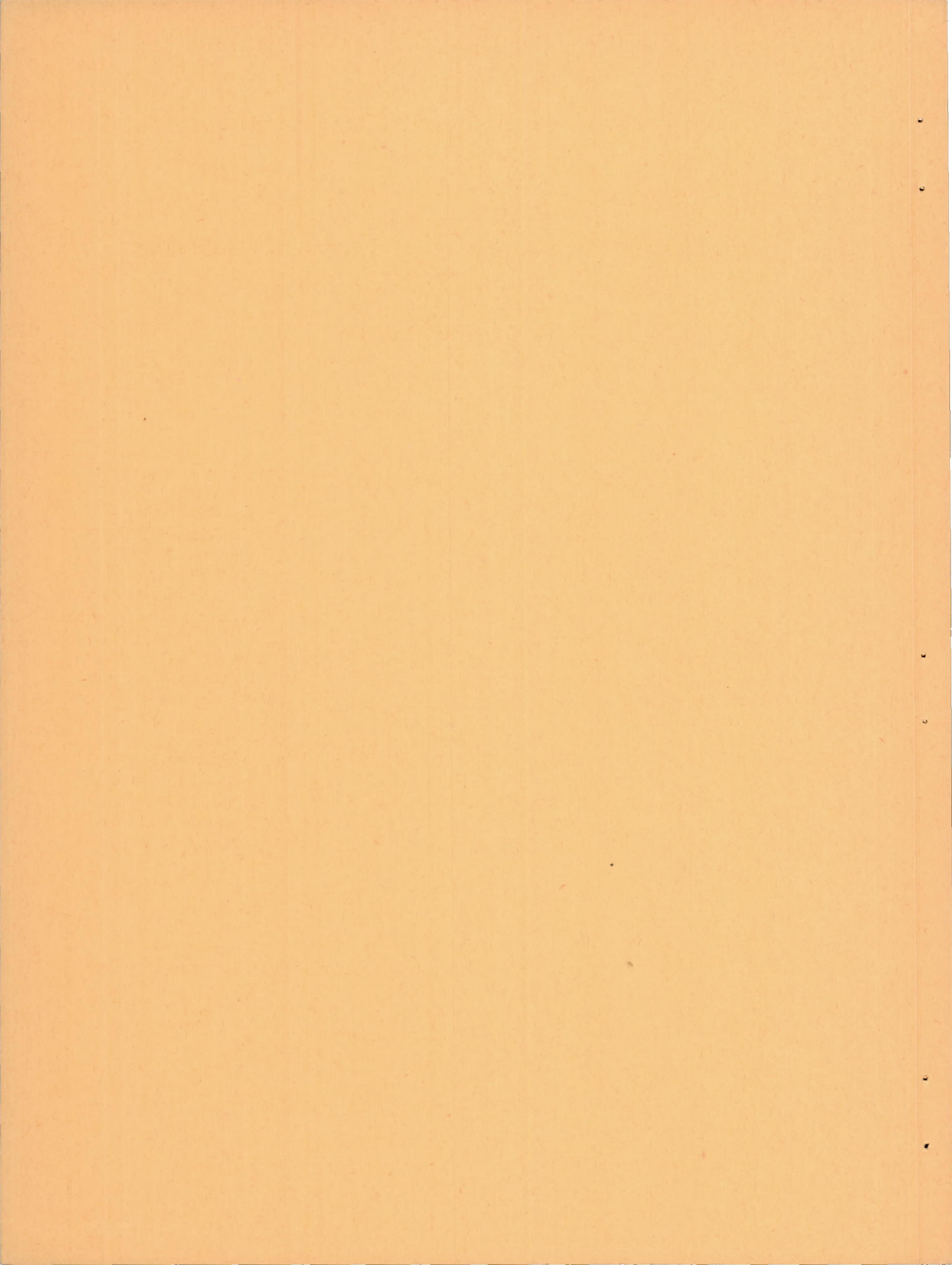


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COMPARISON OF CRYSTAL STRUCTURES OF 10 WROUGHT HEAT-RESISTING ALLOYS AT ELEVATED TEMPERATURES WITH THEIR CRYSTAL STRUCTURES AT ROOM TEMPERATURES

By J. Howard Kittel

SUMMARY

An investigation was made to compare the crystal structure of the predominant phase at temperatures of 1200°, 1500°, and 1800° F in 10 wrought heat-resisting alloys with the crystal structure observed in the alloys at room temperature. The following alloys were investigated: S-816, S-590, Hastelloy B, 19-9 W-Mo, N-155, 16-25-6, K-42-B, Inconel X, Nimonic 80, and type 347 stainless steel. After specimens of each alloy were maintained at the various elevated temperatures for 1 hour, observations were made of the crystal structure of the alloy at the elevated temperatures, using a Geiger-counter X-ray spectrometer modified for high-temperature X-ray diffraction studies. The alloy 19-9 W-Mo, when heated, began to transform from a two-phase mixture of body-centered and face-centered cubic structures to a single phase of face-centered cubic structure between 1400° and 1450° F. The transformation was completed between 1450° and 1500° F. The other nine alloys retained room-temperature crystal structure up to a temperature of 1800° F. A study of the thermal-dilatation characteristics of the alloys up to 2150° F was also made. None of the alloys, including 19-9 W-Mo, showed discontinuities in their dilatation curves that were ascribable to a phase transformation.

INTRODUCTION

The efficiency of gas turbines may be raised by increasing the operating temperature of the working fluid. Current gas turbines are, however, required to operate below optimum temperatures because of limitations in elevated-temperature physical properties of the materials from which the turbine parts are fabricated.

The gas-turbine wheel, the buckets, and the combustion-chamber liners are the most frequent sources of failure in aircraft gas turbines. Increases in operating temperatures or stresses accelerate the rate of failure occurring in these critical parts of the turbine.

The physical properties of alloys at elevated temperatures are dependent to a large extent upon the stability of the alloys at these temperatures. If the primary phase in the alloy should undergo a change in crystal structure, for example, when heated to the operating temperature of the turbine, some modification in the physical properties of the alloy would very likely occur with immediate effect upon the service life of the alloy in the turbine.

In order to detect the possible occurrence of phase changes at elevated temperatures in certain heat-resisting alloys, an investigation was made of the crystal structure at elevated temperatures and at room temperature of 10 wrought alloys currently used in gas turbines. The crystal structure at room temperature of some of the alloys had been previously determined (reference 1). Data are presented in this paper from X-ray diffraction studies of the alloy crystal structures and from thermal-dilatation studies of the alloys. The X-ray diffraction data were obtained with apparatus built especially for elevated-temperature X-ray diffraction studies. Acknowledgement is made to Dr. H. Friedman and to Mr. L. S. Birks of the U. S. Naval Research Laboratory, Anacostia Station, Washington, D. C. for their suggestions concerning the design of the apparatus.

The 10 alloys investigated were S-816, S-590, Hastelloy B, 19-9 W-Mo, N-155, 16-25-6, K-42-B, Inconel X, Nimonic 80, and type 347 stainless steel.

APPARATUS AND PROCEDURE

Specimens for both the X-ray diffraction investigation and the thermal-dilatation investigation were taken from round bar stock. The X-ray specimens measured 1 by 1/2 by 1/8 inch and the dilatation specimens, all $2\frac{1}{2}$ inches in length, ranged from 1/2 to 1 inch in diameter. Nominal chemical compositions of the 10 alloys are listed in table I.

X-ray diffraction patterns of the 10 alloys were made on a Geiger-counter X-ray spectrometer using Fe K α radiation. The

spectrometer was modified as shown in figure 1 to enable X-ray diffraction studies of specimens at elevated temperatures under non-oxidizing conditions. This modification was accomplished by replacing the specimen holder that was supplied with the spectrometer with the apparatus shown in figure 2.

The specimen was heated by a coil of tungsten wire embedded in beryllium oxide. In order to maintain a specimen temperature of 1800°F , a heater input of about 370 watts was required. A thermocouple was welded on the face of the specimen just below the X-ray beam. The heater was supported by a ceramic rod to minimize heat conduction to the base of the chamber. The lower end of the supporting rod was contained in a centering flange that enabled the specimen to be centered accurately in the X-ray beam and with respect to the Geiger counter. The specimen was surrounded by two radiation shields; the one nearer the specimen was nickel foil and the outer shield aluminum foil. The thermocouple wires and the electrical leads to the heater passed through the base of the chamber, which was water-cooled. The wall of the chamber contains two windows of 0.010-inch celluloid for passage of the X-ray beam. The chamber was evacuated to about 10^{-4} millimeters of mercury by an oil diffusion pump attached by a $1\frac{1}{4}$ -inch flexible metal hose to the outlet above the windows. Vacuum tight, demountable joints between the wall and the top and the base of the chamber were effected by the use of 1/16-inch synthetic rubber gaskets.

Diffraction patterns were obtained with the Geiger counter moving at such a rate that θ , the glancing angle of the X-ray beam, changed at a rate of 0.5° per minute. The output of the Geiger tube was automatically recorded as a function of θ . X-ray diffraction patterns of the 10 alloys were obtained for values of 2θ between the angles of 10° and 80° at temperatures of 70° , 1200° , 1500° , and 1800°F . This range of temperatures includes the normal operating temperatures of the critical parts of current gas turbines. Before the specimen was heated, a room-temperature X-ray diffraction pattern of each alloy was recorded. After specimens of each alloy were maintained at the various elevated temperatures for 1 hour, diffraction patterns were recorded at the elevated temperatures. After permitting the specimens to cool, room-temperature patterns were again recorded.

A recording dilatometer with a magnification ratio of 110 was used to obtain dilatation curves of the alloys. The specimens were heated at a rate of 20°F per minute up to 1000°F , and at

5° F per minute above that temperature. Several dilatation curves of each alloy were made. These data were obtained to supplement the X-ray data in the event that any of the alloys were subject to a phase change that would produce a discontinuity in its thermal-expansion curve.

RESULTS AND DISCUSSION

The interplanar spacing values of the diffracted lines obtained from the primary solid solution of each alloy are listed in table II for each temperature investigated. With Fe K α radiation, reflections from only the (111) and (200) planes of the face-centered-cubic primary solid solution in the alloys fall in the range of 2θ from 10° to 80°.

All alloys showed a shift in the angles of diffracted lines because of normal thermal expansion of the crystal lattice. Although the spectrometer is unsuited for accurate measurement of lattice parameters, it could be readily determined that at 1800° F the lattice of each alloy had expanded about 2 percent over its room-temperature value.

One of the alloys, 19-9 W-Mo, showed a change in its crystal structure at a temperature between 1200° and 1500° F. At 70° and 1200° F the alloy showed a line from the (110) planes of a body-centered cubic phase in addition to lines from the (111) and (200) planes of a face-centered cubic phase. The intensity of the (110) line was about one-fifth that of the (111) line. At 1500° and 1800° F diffraction lines from only the face-centered cubic phase appeared. An investigation was then made of the crystal structure of the alloy at 50° intervals between 1200° and 1500° F. When heated, the two-phase structure began to transform to a one-phase structure between 1400° and 1450° F. The transformation was completed between 1450° and 1500° F.

With the exception of the alloy 19-9 W-Mo, all the alloys investigated maintained a face-centered cubic structure after 1 hour at temperatures up to 1800° F, probably because of the high nickel content. Nickel, a face-centered cubic metal, strongly stabilizes the face-centered cubic gamma phase in iron and the face-centered cubic beta phase in cobalt (reference 2).

The alloy 19-9 W-Mo nominally contains only 0.1 percent carbon with an excess of the carbide-forming elements tungsten, molybdenum, columbium, and titanium. Any excess of these four elements after

carbide formation will tend to stabilize the body-centered cubic alpha phase in iron. Because the alpha phase is present in detectable amounts in the alloy at 1450° F and lower, the nickel content apparently is insufficient to counteract the effect of the excess carbide-forming elements.

A typical dilatation curve for each alloy is shown in figure 3. None of the 10 alloys showed a discontinuity in thermal expansion at temperatures up to 2150° F, the highest temperature investigated. It is of interest that the phase change occurring in the alloy 19-9 W-Mo did not noticeably affect its dilatation characteristics.

SUMMARY OF RESULTS

The following results were noted in an investigation of the crystal structure at 70°, 1200°, 1500°, and 1800° F of the 10 wrought heat-resisting alloys S-816, S-590, Hastelloy B, 19-9 W-Mo, N-155, 16-25-6, K-42-B, Inconel X, Nimonic 80, and type 347 stainless steel:

1. The alloy 19-9 W-Mo when heated began to transform from a two-phase mixture of body-centered and face-centered cubic structures to a single phase of face-centered cubic crystal structure between 1400° and 1450° F. The transformation was completed between 1450° and 1500° F.
2. The other nine alloys investigated retained their room-temperature crystal structure at each temperature studied.
3. Thermal-dilatation curves of all the alloys investigated showed no discontinuities in thermal expansion at temperatures up to 2150° F.

Flight Propulsion Research Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, August 12, 1947.

REFERENCES

1. Kittel, J. Howard: The Crystal Structure at Room Temperature of Eight Forged Heat-Resisting Alloys. NACA TN No. 1102, 1946.
2. Hansen, Max: Der Aufbau der Zweistofflegierungen. Julius Springer (Berlin), 1936, pp. 697, 498.

TABLE I - NOMINAL CHEMICAL COMPOSITIONS OF 10 WROUGHT
HEAT-RESISTING ALLOYS

Alloy	Nominal chemical composition, percent									
	C	Cr	Ni	Co	Mo	W	Cb	Ti	Fe	Other
S-816	0.4	20	20	44	4	4	4		3	
S-590	.4	20	20	20	4	4	4		25	
Hastelloy B	.1		64		29				5	
19-9 W-Mo	.1	19	9		.4	1.3	.4	.4	68	
N-155	.3	20	20	20	3	2	1		32	N 0.1
16-25-6	.15	16	25	6					51	N .2
K-42-B	.05	18	42	22				2.2	14	Al .2
Inconel X	.05	15	73				1	2.5	7	
Nimonic 80	.05	21	75					2.5		Al .6
Type 347 stainless steel	.05	18	10				.5		70	

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TABLE II - SPACINGS OF (111) AND (200) PLANES OF 10 WROUGHT
HEAT-RESISTING ALLOYS AT ELEVATED TEMPERATURES

Alloy	Temperature, °F			
	70	1200	1500	1800
	Interplanar spacing, d, Å			
S-816	2.03	2.04	2.05	2.06
	1.76	1.78	1.79	1.79
S-590	2.04	2.05	2.06	2.07
	1.77	1.79	1.79	1.80
Hastelloy B	2.04	2.06	2.07	2.09
	1.78	1.79	1.80	1.81
19-9 W-Mo	2.06	2.07	2.07	2.08
	2.02 ^a	2.02 ^a	-----	-----
	1.78	1.80	1.80	1.82
N-155	2.06	2.07	2.07	2.09
	1.79	1.80	1.80	1.82
16-25-6	2.07	2.09	2.10	2.10
	1.80	1.81	1.82	1.83
K-42-B	2.05	2.06	2.07	2.07
	1.78	1.79	1.79	1.80
Inconel X	2.03	2.04	2.05	2.06
	1.76	1.78	1.78	1.79
Nimonic 80	2.05	2.05	2.06	2.07
	1.77	1.78	1.79	1.80
Type 347 stainless steel	2.06	2.07	2.08	2.10
	1.78	1.80	1.80	1.82

^aInterplanar spacing for (110) plane from a body-centered cubic phase that does not exist in the alloy at the two higher temperatures.

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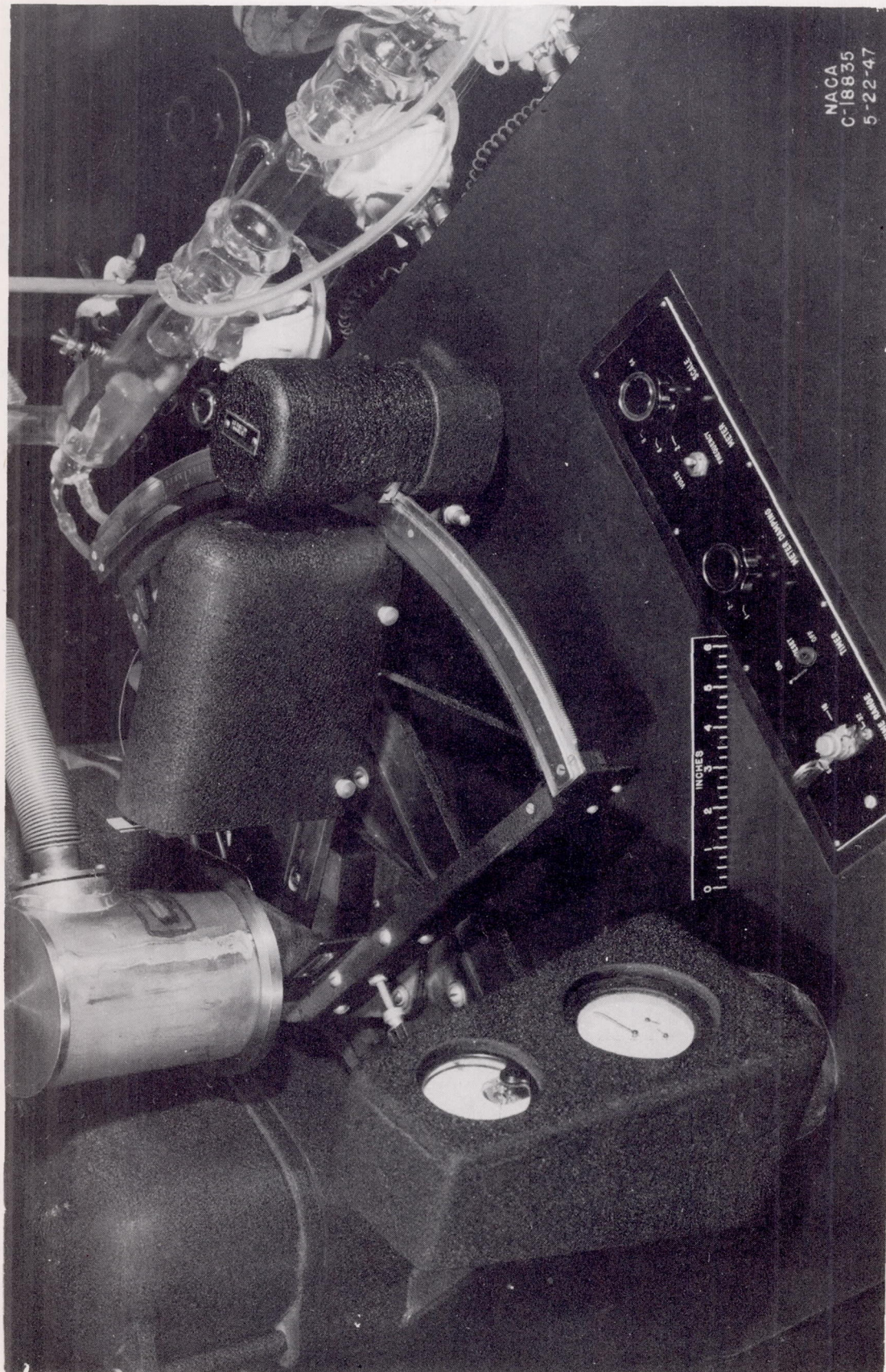


Figure 1. - High-temperature X-ray diffraction apparatus.

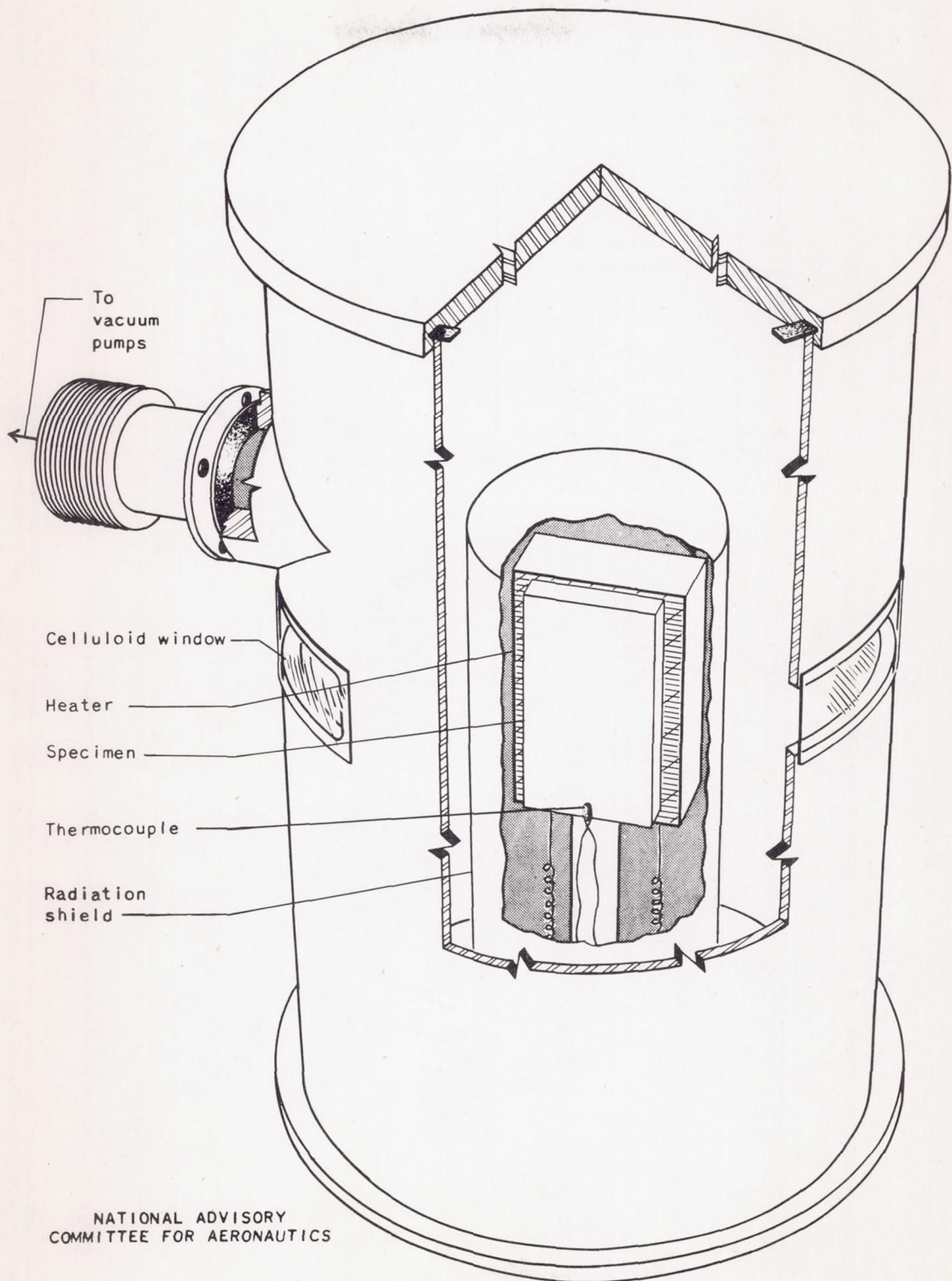
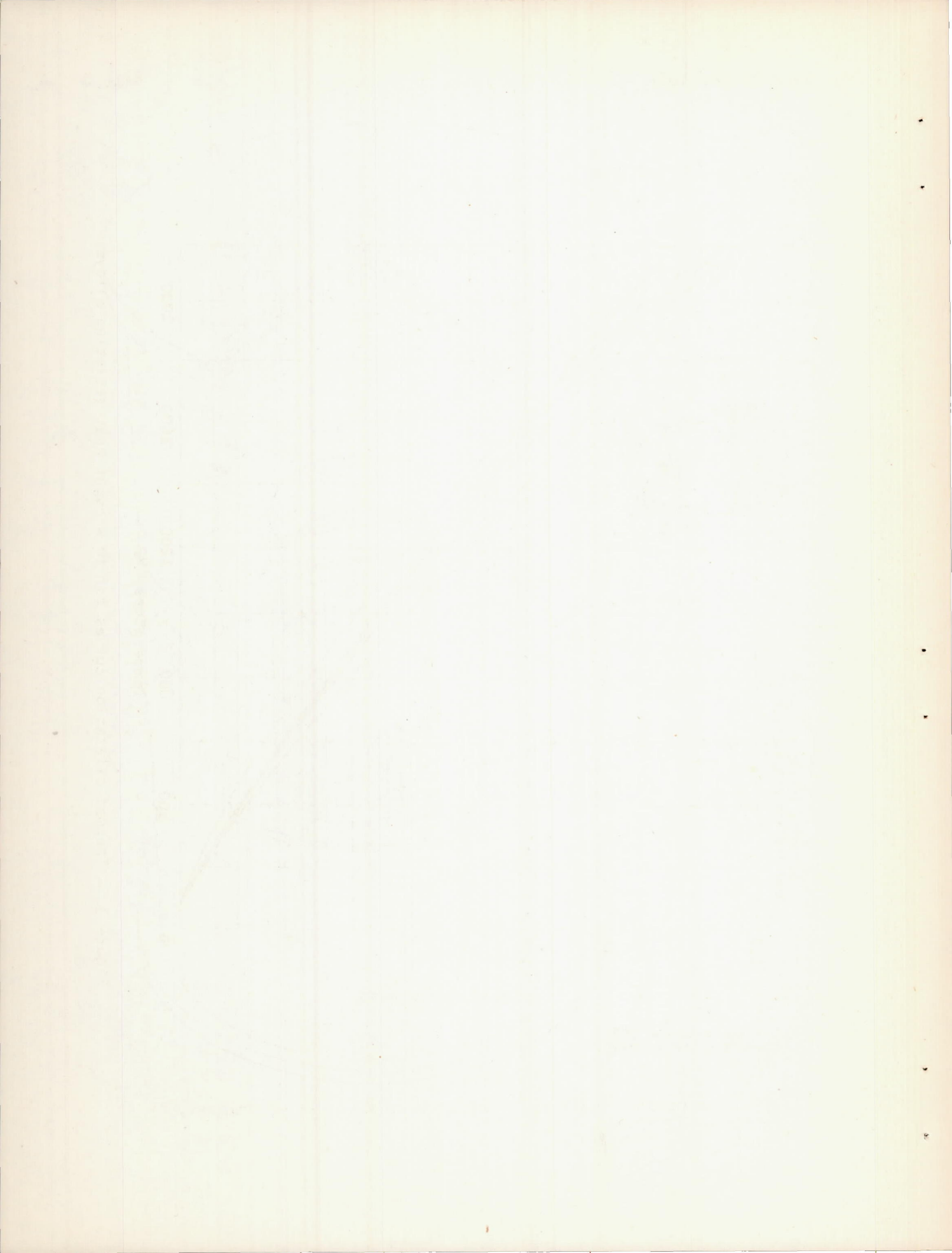


Figure 2. - Sectional view of specimen heating chamber.



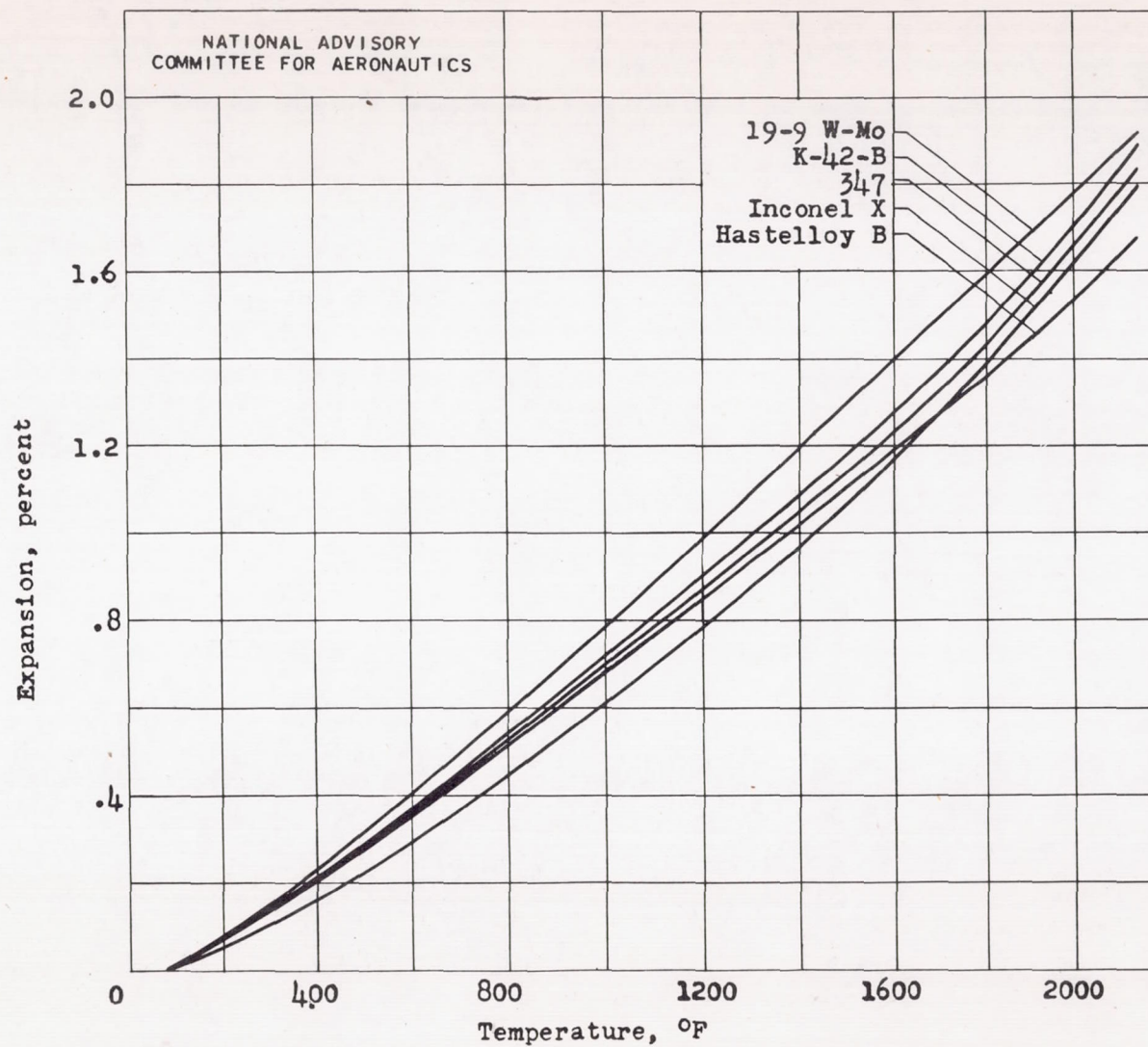


Figure 3. - Thermal-dilatation curves for 10 wrought heat-resisting alloys.

